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**Carderock Division
Naval Surface Warfare Center**

Bethesda, Md. 20084-5000

CARDIVNSWC-SSM-61-93/18 July 1993
Survivability, Structures, and Materials Directorate
Technical Report

**High Temperature Deformation Behavior of
YBa₂Cu₃O_{6+x} Superconducting
Ceramic Materials**

by
A. S. Rao, L. F. Aprigliano and O.P. Arora

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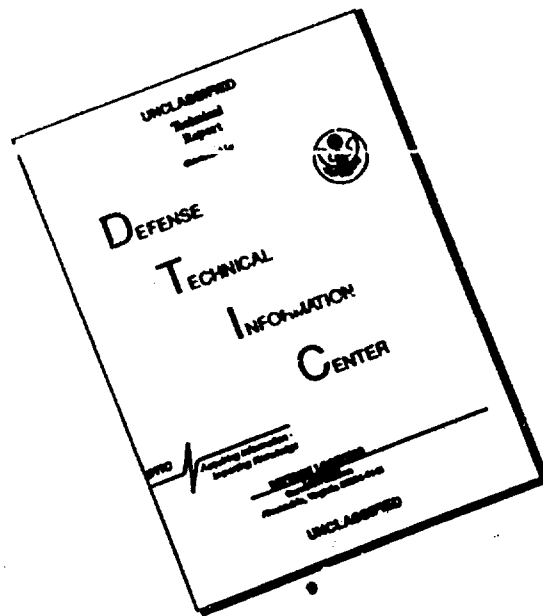
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ABSTRACT

Superplastic deformation is being explored as a means to turn the new ceramic superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ into useful Naval hardware. Maximum superplastic deformation can occur when the grain size and deformation temperature are optimized. In order to obtain optimum size, three additives (alumina, silver oxide and silver) were studied. Deformation temperatures were selected in the range 23 - 850°C. The results indicate that while alumina is very effective in regulating the grain size, it has a devastating effect on the deformation mode. Both silver and silver oxide have no effect on the grain size. However, they tend to improve the superconducting properties of $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$.

The deformation experiments that were conducted on commercially available, fine $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ powders suggest that the fine ceramic undergoes superplastic deformation above 800°C. The maximum deformation that can be achieved without the degradation of superconductivity is about 62 %. However, this deformation depends upon a slow strain rate and a temperature of 850°C. The addition of silver improves the maximum deformation that the composite can sustain without the loss of the superconducting property. A maximum deformation of about 110 % was achieved in silver composites that were deformed at a slow strain rate of 1.7×10^{-4} inch per inch per second at 850°C.

ADMINISTRATIVE INFORMATION

This report was funded by the CDNSWC Independent Research Program, sponsored by the Office of the Naval Research, ONR 10, and administered by the Research Director, CDNSWC 0112, Dr. Bruce Douglas, under program element 61152N, Task Area ZR-000-01-01 and CDNSWC Work Unit 1-2812-148. This work was supervised within the Metals and Welding Department (Code 612) by Dr. O. P. Arora.

INTRODUCTION

Background

The advancement of superconducting ceramic materials with zero resistance above liquid nitrogen temperature [1,2] has prompted a race for fabricating these brittle ceramics into useful components. Although the fabrication of superconducting ceramic thin films has been explored using various techniques, such as ion beam deposition and vapor phase deposition etc. [3-6], no significant advancement in the processing of bulk ceramics has been achieved to date. However, some investigators have suggested that if during the initial phase of component processing a suitable metallic species (for example gold or silver) is added to the hard and brittle ceramic system (such as superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$), the final composite can exhibit an improvement in its ductility. This is because the addition of noble metals and silver does not alter the chemical stoichiometry of the $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ ceramic material and its superconducting properties. In addition, the above metals (in particular the silver) improve the critical current carrying capacity [7], lower the normal state resistivity [8], improve the mechanical strength and fracture toughness [9] and reduce the adverse affect of water in the system [10].

For large scale manufacturing, the processing based on the utilization of noble metals or silver may not be cost effective. However, the improvement in the superconducting properties and the material formability may justify to outweigh the cost. This project was undertaken in order to ascertain the advantage of the

addition of silver during processing on the formability of the superconducting materials.

Superplasticity

Superplasticity is defined as the ability of certain materials to undergo unusually large amounts of plastic deformation before local necking or failure occurs [11]. Since the discovery of superplasticity in brass [12], a great number of metal systems have been found to undergo superplastic flow [13]. During the last two decades, this phenomena was observed in many ceramic systems, including pure and doped zirconia, alumina, glass ceramics, magnesia spinel, barium titanate, and silicon carbide [14-19]. This work on these superconducting ceramics contradicts the concept that room temperature ductility is a necessary condition for superplastic flow.

In general, the strain rate, $\dot{\epsilon}$, during creep or superplastic deformation depends upon the grain size, and can be given by the expression [20]

and
$$\dot{\epsilon} = k \sigma^n (\bar{d})^{-p} \exp (-Q/RT) \quad \dots\dots\dots (1)$$

$$\sigma = k (d\epsilon/dt)^m \quad \dots\dots\dots (2)$$

where σ is the flow stress and ϵ is the strain
 $\dot{\epsilon} = (d\epsilon/dt)$ is the strain rate
 k is a constant and lies between 0.3 - 1 for most of the superplastic materials [7]
 n is the stress exponent
 m is the strain rate sensitivity
 \bar{d} is the grain size
 $p = 2$ for lattice diffusion and $p = 3$ for grain boundary diffusion
 Q is the activation energy
 R is the gas constant and
 T is the deformation temperature

If it is assumed that $n = 1$ for the superplastic deformation of $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ superconductor, then at a given strain rate and temperature [21], the stress is then dependent on the inverse square (or cube) of the grain size. If the grain size is reduced by a factor of 10, then the flow stress decreases by 100 (or 1000) times. This illustrates the dramatic impact of the grain size on superplasticity.

The purpose of this study is to examine the conditions under which superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ ceramic materials can be superplastically deformed at temperatures that are below the sintering temperature. In this report, the processing methodology and high temperature deformation results obtained from superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ ceramic samples with and without additives are being reported.

EXPERIMENTAL

The general prerequisites for superplasticity are fine grain size (typically 100 - 200 nm), high density, and low strain rate. The microstructure should be controlled to meet the first and second conditions. However, it is not possible to satisfy all the above three conditions simultaneously by the present sintering technology. So the conditions were chosen to get higher density rather than smaller grain size with clean microstructure, or to get smaller grain size and a reasonable density.

Both as-synthesized and commercial superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ powders were used during the present investigation. While the as-synthesized powder was produced from Y_2O_3 , BaCO_3 and CuO using the

solid state chemical reaction method, the commercial powders were synthesized using both the solid state chemical reaction and chemical coprecipitation methods. The details of the powder processing were given elsewhere [22]. Three additives (alumina, silver oxide and silver with average particle size ~ 1 micrometer) were used in the present investigation and the concentration of the additive range from 0 - 25 vol.% (0 - 38 wt.%). Predetermined amounts of as-synthesized $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ powder and the additive (alumina or silver oxide or silver) were mechanically mixed and were ground for 60 minutes in a ball mill using zirconia balls. Both pure $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ powder and the powder mixture containing either alumina or silver oxide or silver were later cold pressed under 25,000 psi. The pressed samples were sintered at 920°C for 2 hours and were annealed at 550°C for 6 hours in flowing oxygen and were slow cooled to the room temperature. The sintered samples were polished and were cut into 2 x 2 x 10 mm bars. These samples were used for particle size, structural and electrical property determination. Since the samples that are used for mechanical deformation require nearly 95 % density, a different sintering procedure was adopted. In this procedure, the powders were isostatically pressed under 50,000 psi and were sintered at 925°C for 6 hours. Later the samples were annealed in flowing oxygen for 12 hours at 500°C .

High Temperature Deformation

The high temperature deformation experiments were carried out at the Lehigh University using their compression test facility. The high temperature compression unit consists of a tensile

testing machine that has been fitted with a furnace unit. The special furnace unit is a split type furnace with a stable heating zone. In order to minimize the shear stresses which may result from the misalignment, a pair of grips were designed which could place the samples in compression while the grips were pulled in tension by the deformation test unit. Figure 1 shows the schematic diagram of the grips used for the high temperature compression test. Because Inconel X 750 has high strength and oxidation resistance at temperatures up to 980°C, it was chosen for, the grips for the deformation testing. The yield strength of the grip material was > 119,000 psi which is much higher than the stress needed for deforming the sample.

The test samples were held between the grips and were heated to the desired test temperature at the rate of 10°C per minute. Once the specimen temperature reached the deformation temperature, the samples were maintained at that temperature for 2 hours for the stabilization of the thermal expansion of the test system. The samples were then compressed at a constant strain rate of either 1.7×10^{-4} inch per inch per sec or 4.2×10^{-4} inch per inch per sec. After the compression test, the furnace was turned off and was cooled to room temperature. The post deformed samples were reannealed in flowing oxygen at 500°C for 12 hours and then their electrical resistance was measured as function of temperature.

Characterization

The particle size distribution and grain morphology of the samples was examined under both optical and scanning electron

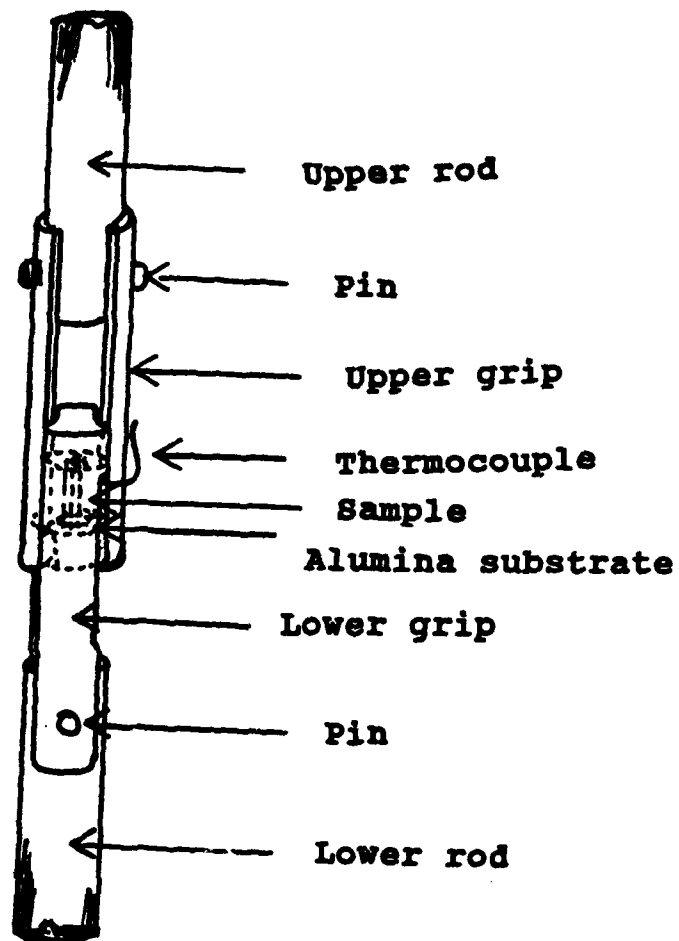


Figure 1. Schematic diagram of the special grips developed for the deformation experiments. The sample is compressed while the grips are pulled in tension.

microscopes. The crystal structure was determined using x-ray diffraction technique. The electrical resistance of all samples was measured using dc four probe electrical resistance method.

RESULTS AND DISCUSSION

The as-synthesized $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ powder has a density of 5.9 gm cm^{-3} and a surface area of $\sim 0.22 \text{ m}^2\text{gm}^{-1}$. The powder is poly-dispersed and has an average particle size of ~ 10 micrometers. The average particle size of the two commercial powders that were used in the present investigation were 5 and 3 micrometers. The detailed analysis of the powder characteristics was given earlier [22]. Figures 2 - 4 show typical morphology of the $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ composites containing different amounts of either alumina, silver oxide or silver respectively. The results suggest that the addition of even 1 wt.% of alumina has a significant effect on the shape and topography of $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ particles. Both silver oxide and silver have no significant effect on the shape and topography of the $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$. In order to derive some semi-quantitative correlation between the (YBaCu) oxide grain size and the additive concentration, some samples were polished and a number of optical micrographs were obtained at random areas that represent the entire surface of the sintered test samples. Figure 5 shows the grain size distribution of the sintered sample as a function of the additive concentration. The results suggest that while the additive alumina reduces the $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ particle size, both silver oxide and silver has very little effect in the concentration range 0 - 25 wt.%.

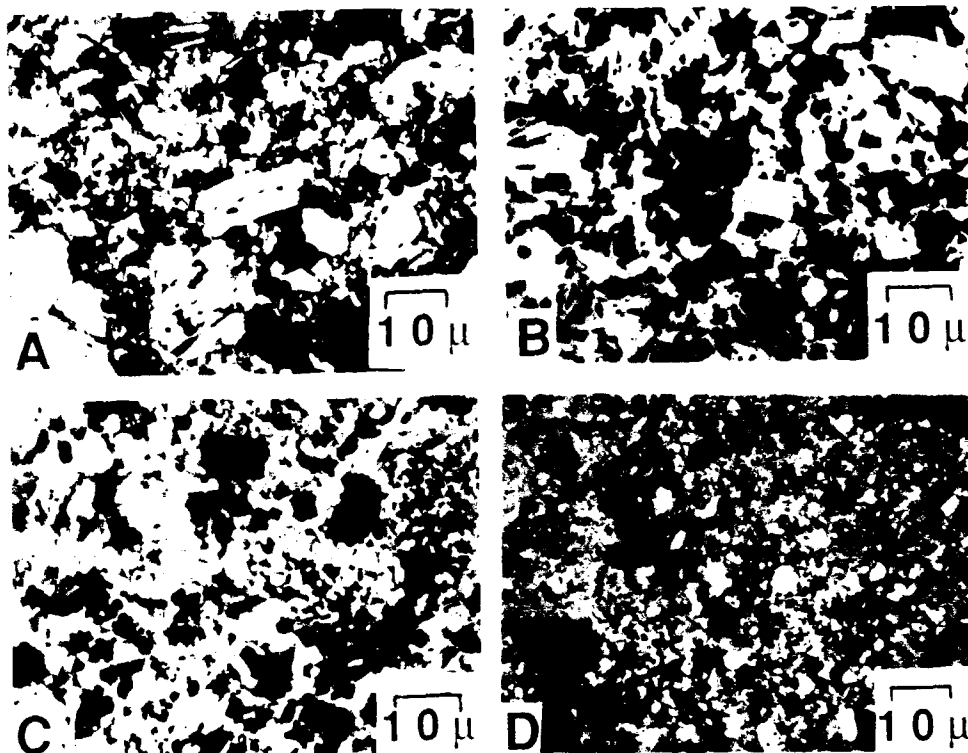


Figure 2. Morphology of aluminum / $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ composites. Aluminum concentration (A) 1, (B) 2, (C) 5 and (D) 15 wt.%.

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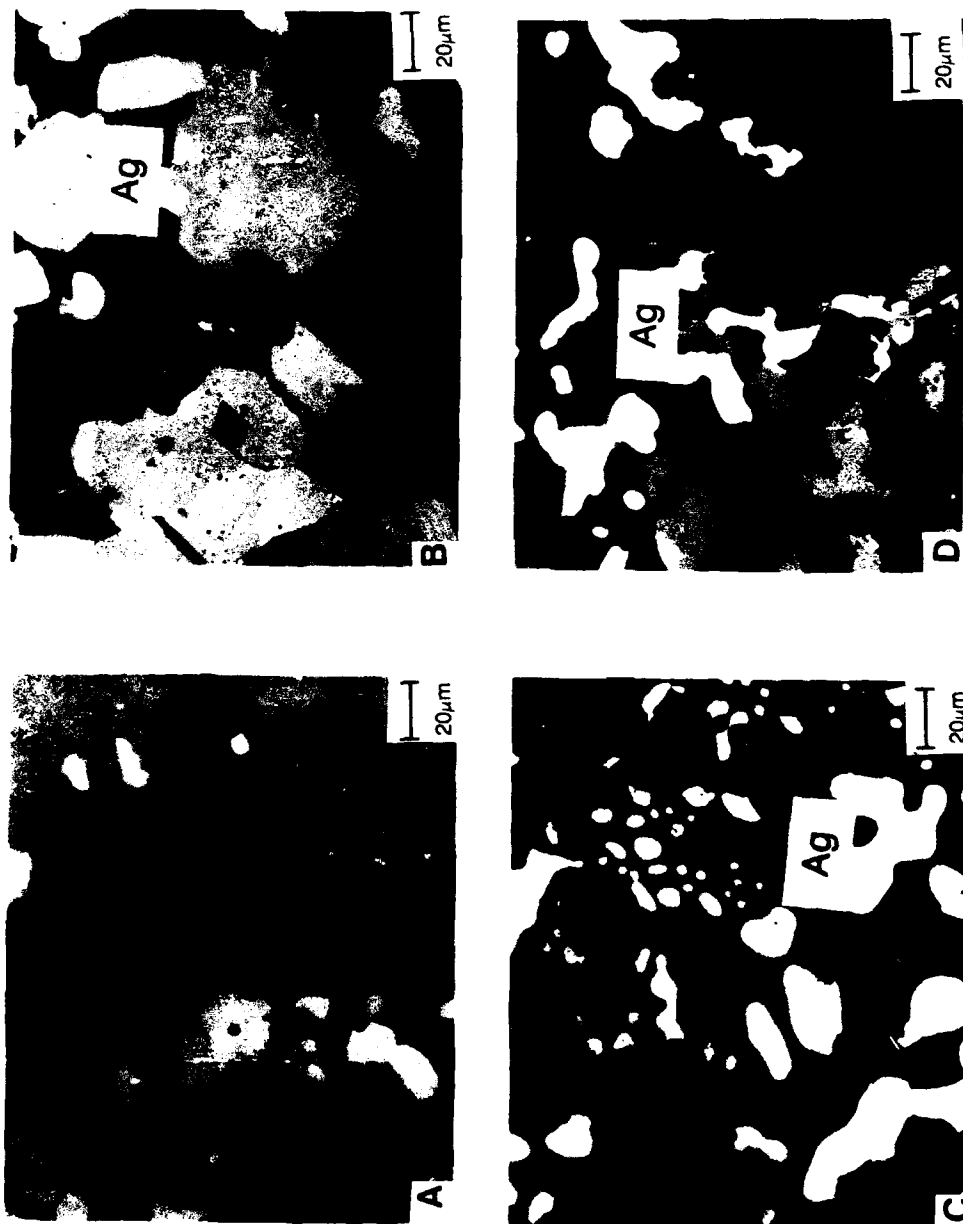


Figure 3. Morphology of silver oxide / $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ composites. silver oxide concentration (A) 0, (B) 2, (C) 5 and (D) 20 wt. %.

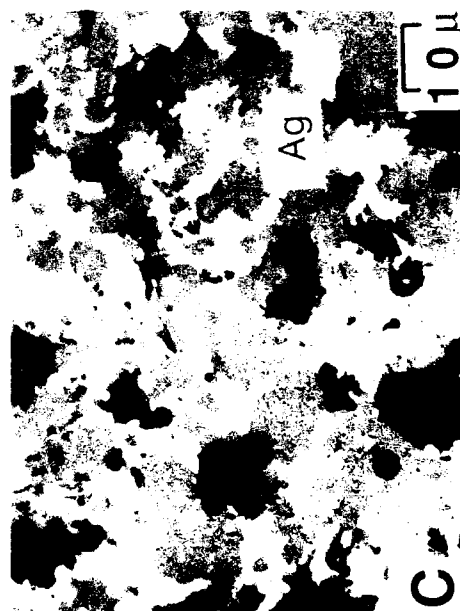
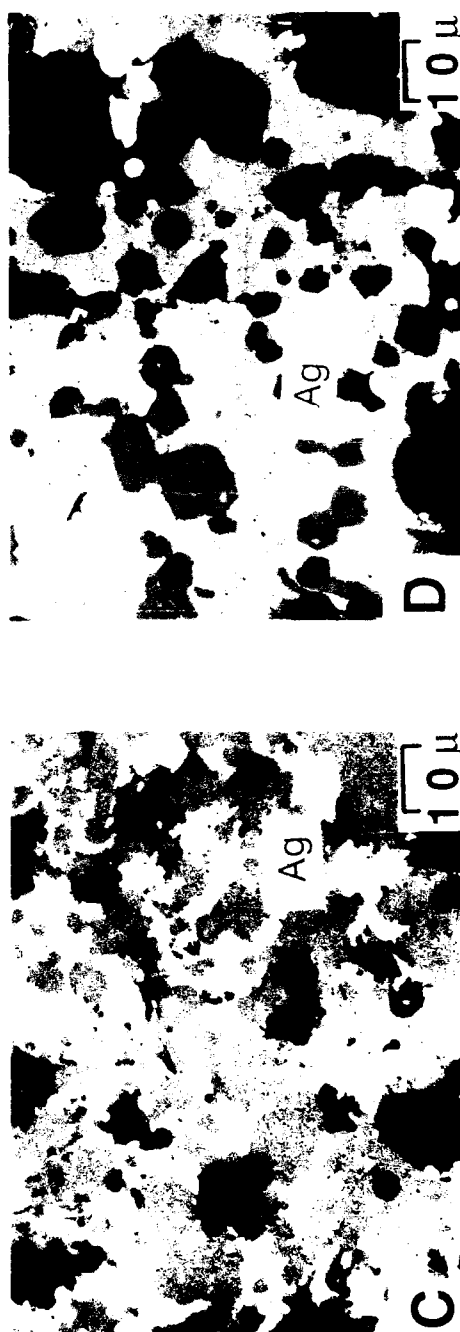


Figure 4. Morphology of silver / $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ composites. Silver concentration (A) 0, (B) 2, (C) 5 and (D) 20 wt.%.

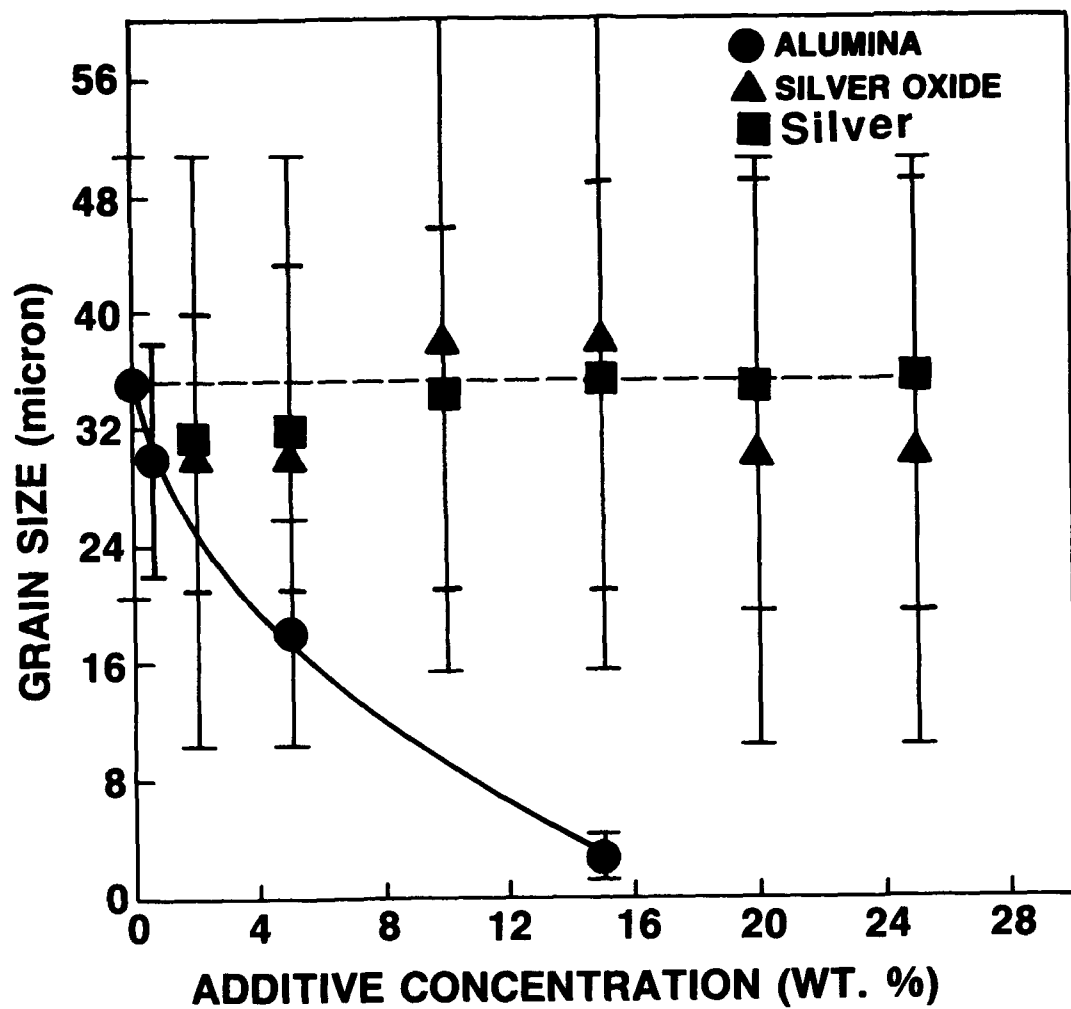


Figure 5. Particle size versus additive concentration profiles of (●) alumina, (▲) silver oxide and (■) silver / $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ composites.

The electrical resistance versus temperature profiles of all the above composites is shown in Figures 6 - 8 respectively. The results suggest that the superconducting transition temperature (T_C) of $YBa_2Cu_3O_{6+x}$ decreases with an increase in the concentration of alumina. Once a critical value (approximately 50 K) is reached, it appears that the addition of alumina in the range of 2 to 10 wt.% has no effect on the T_C . The addition of silver oxide or silver on the other hand tends to improve the T_C of $YBa_2Cu_3O_{6+x}$.

Based on the additive large grain size measurements, it was decided that the as-synthesized $YBa_2Cu_3O_{6+x}$ and the silver oxide or silver / $YBa_2Cu_3O_{6+x}$ composites were not likely to exhibit any plasticity in deformation studies. Therefore in the first set of experiments, the high temperature deformation was carried out on alumina composites containing 5 wt.% alumina.

For deformation studies, only the composites that were isostatically pressed under 50,000 psi and sintered at 925°C for 6 hours and annealed in oxygen at 500°C for 12 hours were used. The samples were subjected to the compression test at a very low strain rate (of 1.7×10^{-4} inch per inch per second). All the test samples failed abruptly by brittle fracture and did not show any plasticity even at 850°C. In the open literature it was reported that pure alumina does not show any plastic deformation below 1100°C.

Therefore it is possible that (i) the deformation temperature used in the present investigation is not sufficient for the composite to show any plastic deformation and (ii) the grain size of as-synthesized $YBa_2Cu_3O_{6+x}$ is too large for deformation experiments.

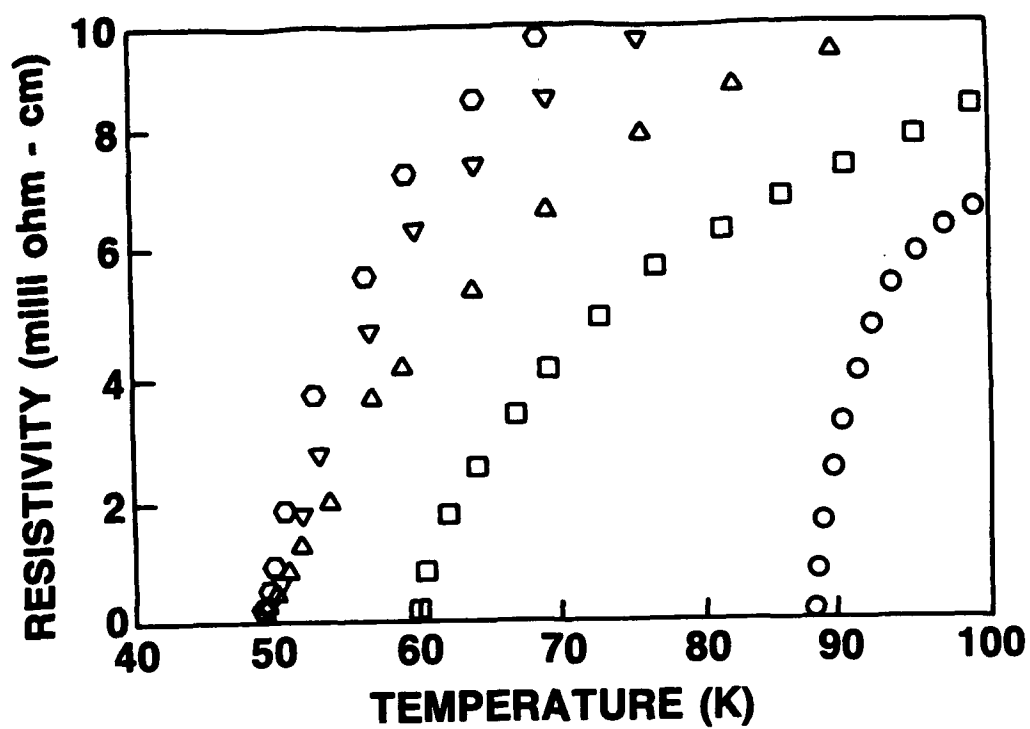


Figure 6. Electrical resistance versus temperature profiles of alumina / YBa₂Cu₃O_{6+x} composites. Alumina concentration (○) 0, (□) 1, (△) 2, (▽) 5 and (⬡) 10 wt.%.

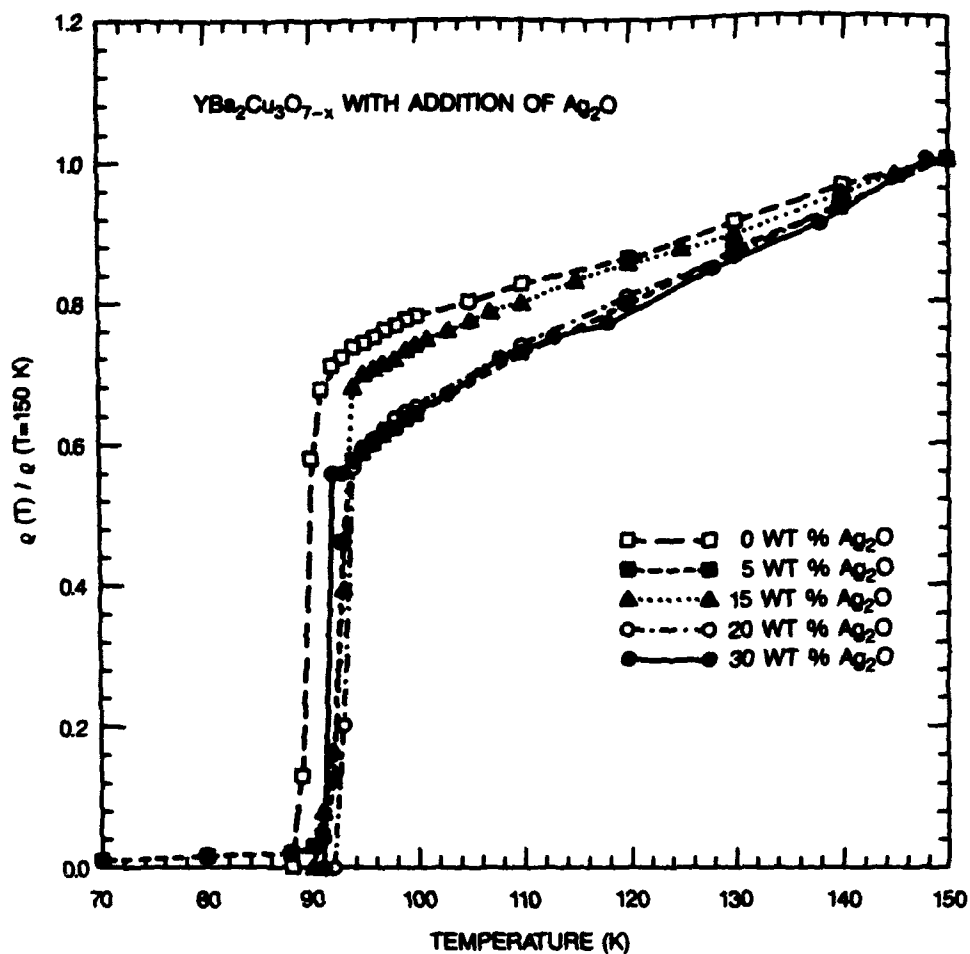


Figure 7. Electrical resistance versus temperature profiles of silver oxide / $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ composites. Silver oxide concentration (□) 0, (■) 5, (▲) 15, (○) 20 and (●) 30 wt.%.

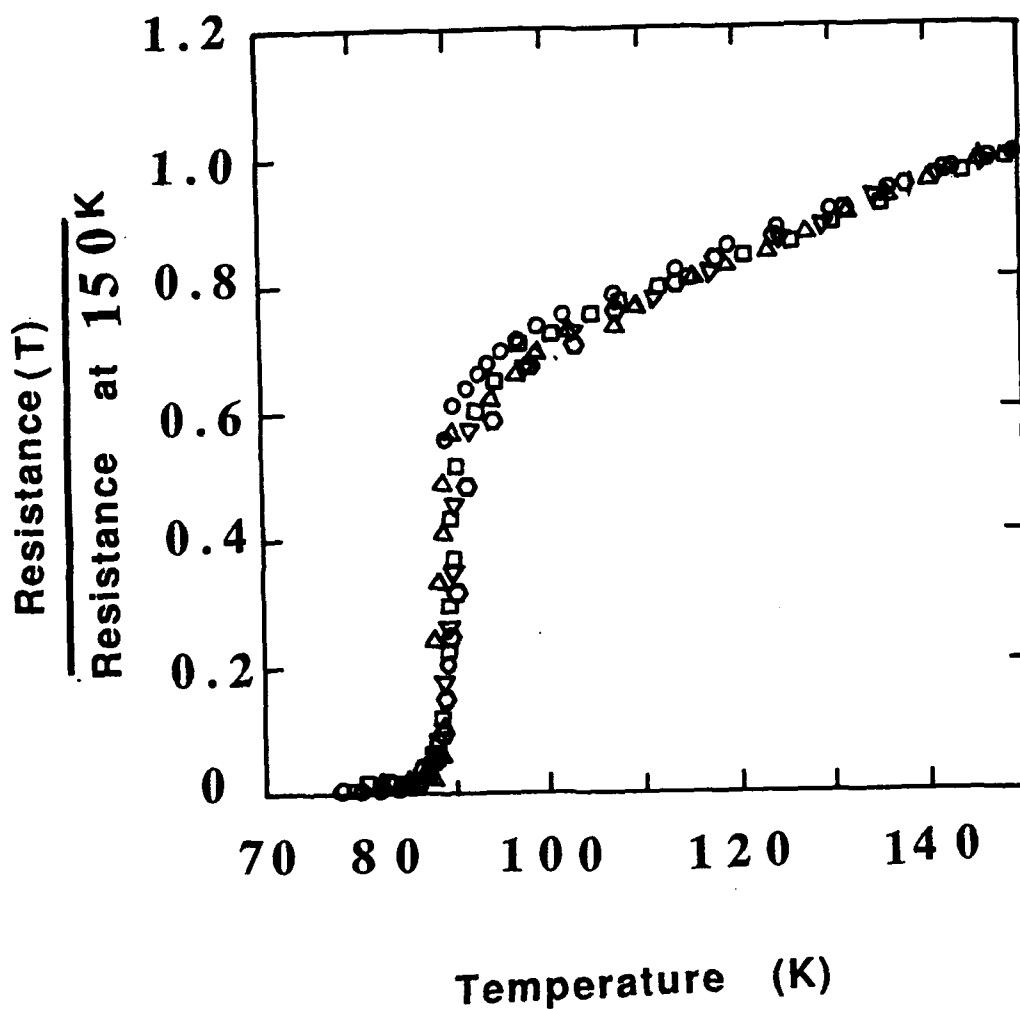


Figure 8. Electrical resistance versus temperature profiles of silver / $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ composites. Silver concentration (○) 0, (□) 2, (△) 5, (▽) 10 and (◇) 20 wt.%.

It has to be pointed out that the maximum deformation temperature was restricted to 850°C because the preliminary experiments indicated that above 850°C, the post - deformed $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ did not show any superconducting property.

In order to establish whether pure $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ shows any plastic deformation, samples that were produced using the 5 micrometer size commercial powder were compressed at a slow strain rate (of 1.7×10^{-4} inch per inch per second) in the temperature range 23 - 700°C. Figure 9 shows typical stress versus strain profiles of pure $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ samples deformed in compression at various temperatures. The results suggest that below 600°C the samples tend to fail in a brittle fracture mode at the end of an initial elastic range. However, they show some plastic behavior when deformed at 700°C. In addition, the results also indicate that the modulus of elasticity decreases with increase in the deformation temperature initially in the temperature range 23 - 450°C. Above 450°C, the modulus increases with temperature (in the range 450 - 600°C). Above 600°C, this trend is reversed. From a number of stress versus strain plots obtained for these $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ samples, it was found that at 700°C pure $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ exhibited ~ 0.6 % plastic deformation.

For superplastic deformation of ceramic materials, the important requirement is that the primary particle size of the sintered composite be as small as possible (typical 100 - 200 nm). All the powders used in the present investigation have larger particle sizes than required. However, 5 micrometer size $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ particles will show some degree of plastic deformation provided they are

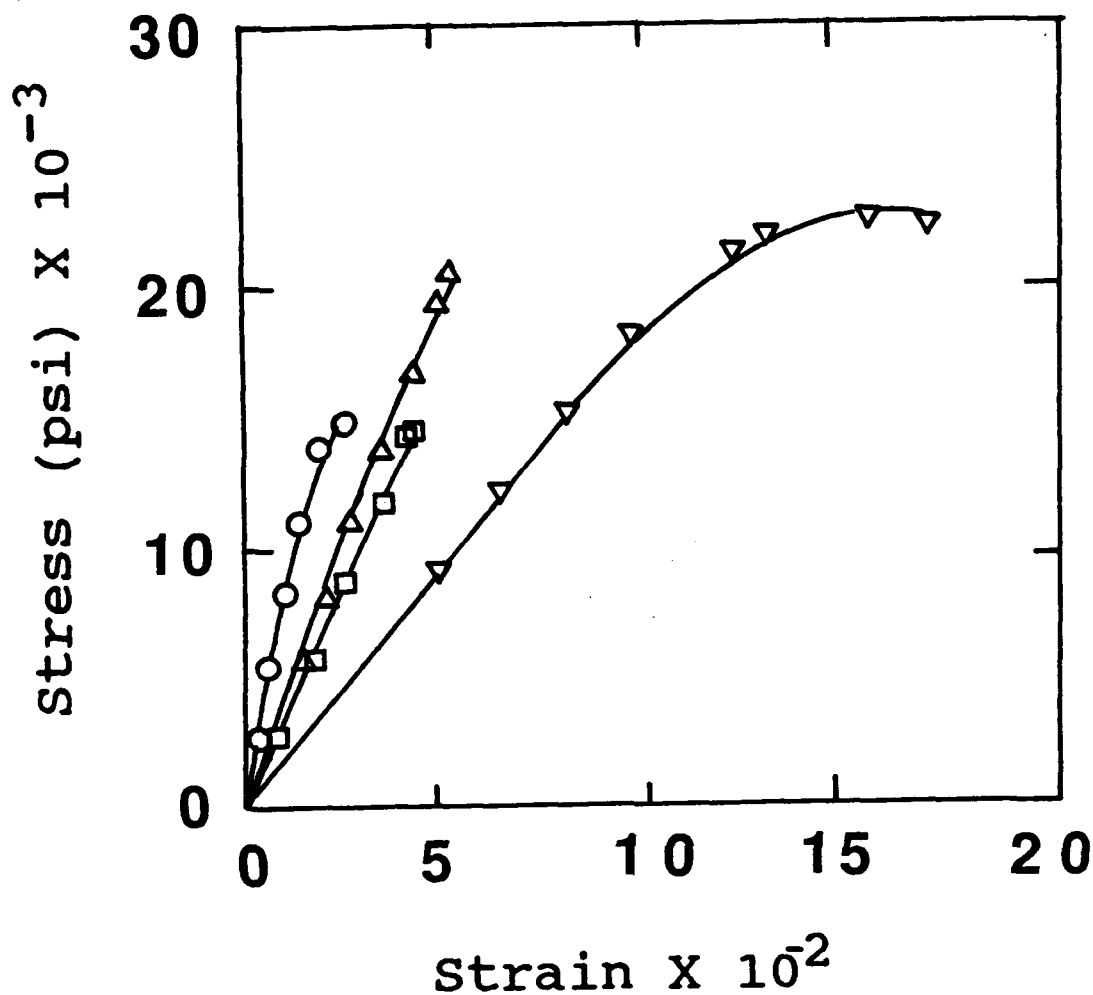


Figure 9. Stress versus strain profiles of pure $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ samples processed using 5 micron size commercial powder. Sample temperature during deformation (○) 23, (□) 450, (△) 600 and (▽) 700°C. Deformation strain rate 1.7×10^{-4} per second.

deformed at high enough temperature. Therefore it is possible that by controlling both the grain size and the deformation temperature an improvement in the degree of plasticity and even superplasticity can be achieved.

In order to ascertain some answers for the above suggestions, two additional experiments were carried out. In the first set of experiments, very fine 3 micrometer size $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ powders were used for the deformation study. In the second set of experiments, 5 and 25 vol.% silver composites were produced with the above powder and those composites were used for deformation. All powders were homogenized and were sintered at 875°C for 6 hours. The sintering temperature was chosen to be 875°C because the low sintering temperature will avoid the grain growth and prevent the formation of liquid phase silver. Typical microstructures of pure $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ and 25 vol.% silver composites are shown in Figure 10. The deformation was carried out on all samples in the temperature range $750 - 800^\circ\text{C}$ and the results are summarized in Table 1. The results shown in Table 1 suggest that at 750°C all samples failed abruptly without showing any significant plastic deformation. At 750°C , the sample with 25 vol.% silver failed slowly without a sharp load drop, implying that the failure mode is not brittle. At 800°C , all samples show significant plastic deformation.

Once it was established that at 800°C the samples did not fail with brittle fracture, it was thought that the onset for plastic flow of the $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ was around 800°C . To evaluate the dependence of deformation mode to the strain rate, some samples were slowly deformed at 800°C initially at the slow strain rate of $1.7 \times$

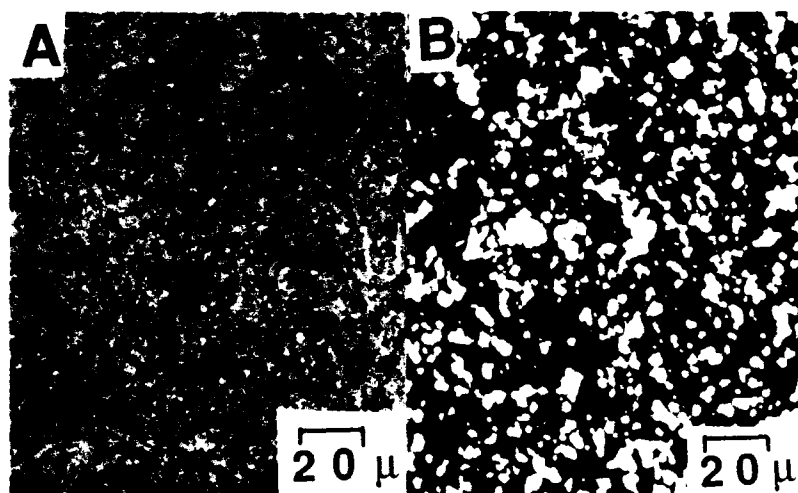


Figure 10. Typical microstructure of pure $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ and 25 vol.% silver composites processed using 3 micron size commercial powder.

10^{-4} inch per inch per second for a period of time and then the strain rate was increased to 4.2×10^{-4} inch per inch per second in one step.

TABLE 1 Summary of the high temperature deformation test of silver / $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ composites.

Silver conc. (vol.%)	Deformation temperature (C)	deformation strain rate (inch per inch per second)	Sample status
0	750	1.7×10^{-4}	Fail
0	750	4.2×10^{-4}	Fail
0	800	1.7×10^{-4}	Plastic flow
0	800	1.7×10^{-4}	Plastic flow
0	800	4.2×10^{-4}	Fail
5	750	1.7×10^{-4}	Fail
5	750	4.2×10^{-4}	Fail
5	800	1.7×10^{-4}	Plastic flow
5	800	4.2×10^{-4}	Fail
25	750	1.7×10^{-4}	Fail
25	750	4.2×10^{-4}	Fail
25	775	4.2×10^{-4}	Fail
25	800	1.7×10^{-4}	Plastic flow
25	800	4.2×10^{-4}	Fail

As a result the stress increased rapidly and the sample failed (Figure 11). Physical examination of the test samples revealed that large shear cracks have developed in the sample due to a sudden increase in the deformation strain rate from 1.7×10^{-4} to 4.2×10^{-4} inch per inch per second. In another experiment, the deformation was carried out at a slow strain rate (of 1.7×10^{-4} inch per inch per

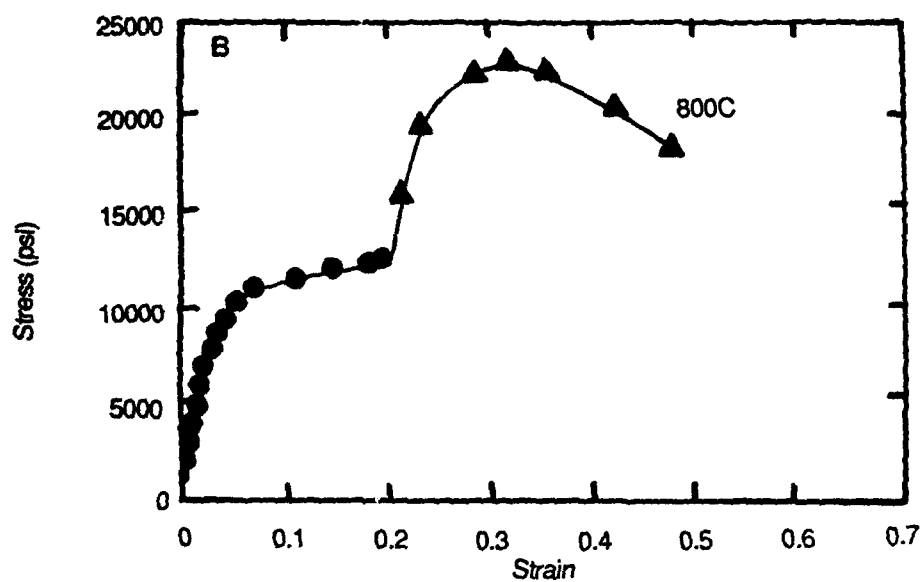


Figure 11. Stress versus strain profiles of sintered $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ samples processed using 3 micron size commercial powder. Deformation strain rate (●) 1.7×10^{-4} and (▲) 4.2×10^{-4} per second. Deformation temperature 800°C .

second), however, the deformation temperature was increased from 800°C to 850°C. In this case the samples did not develop any strain hardening but showed a nearly flat flow stress plateau (Figure 12) and the test sample dimensions were reduced under compression with ~62 % reduction (Figure 13). These results indicate that for the onset of superplastic deformation of $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ the strain rate must be 1.7×10^{-4} inch per inch per second or less.

The silver composites were then tested as a function of both deformation strain rate and deformation temperature. All composite samples showed large plastic deformation (Figure 14). However, the degree of the plasticity depended upon the deformation conditions. For example, the samples that were deformed at 750°C showed a total deformation before the failure of ~ 8%. At 775°C, the maximum deformation that the composites sustained without any visible surface cracks was found to be ~ 46%. At 825°C, the deformation was nearly 70% at an initial strain rate of 5.33×10^{-5} per second. At 850°C, the total deformation of ~ 110% was observed and the deformation remained independent of the strain rate in the range $1.7 - 4.78 \times 10^{-4}$ inch per inch per second.

The important observation in these samples is that the superplastic deformation behavior in $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ appears to be different from that of the superplastic deformation behavior of other ceramic materials. Most ceramic materials when subjected to superplastic deformation undergo significant changes in the grain texture, morphology, and grain size in order to accommodate large shear stress. However, the present $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ and silver composite

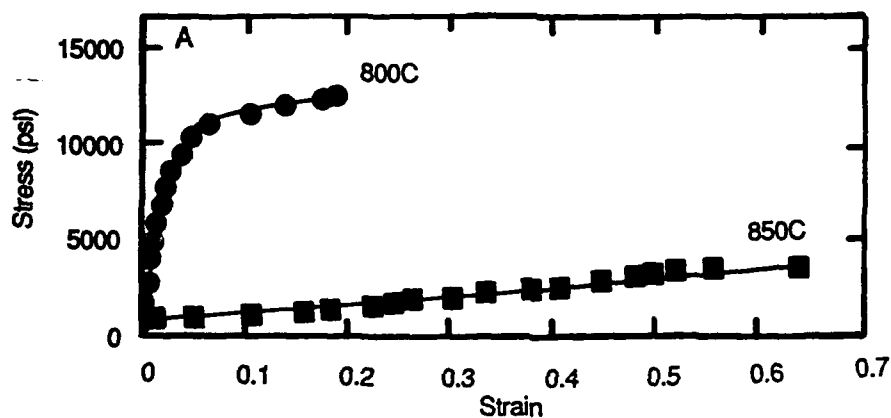


Figure 12. Stress versus strain profiles of sintered $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ samples processed using 3 micron size commercial powder. Deformation strain rate 1.7×10^{-4} per second. Deformation temperature (●) 800 and (■) 850°C.

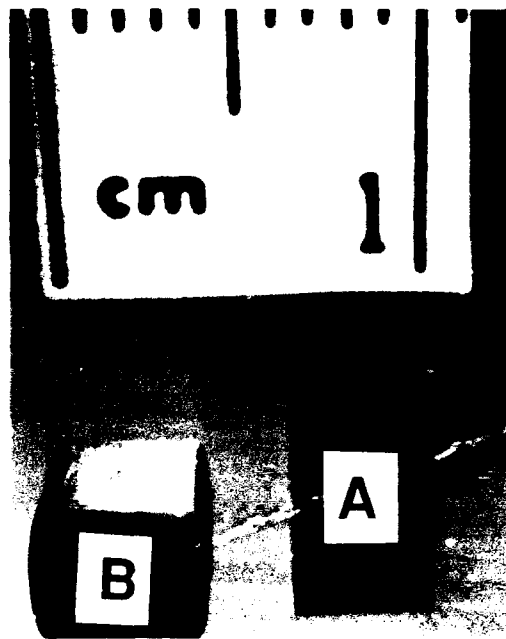


Figure 13. Typical example of the dimensional changes in $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ samples (A) before and (B) after deformation at 850°C . Deformation strain rate 1.7×10^{-4} per second.

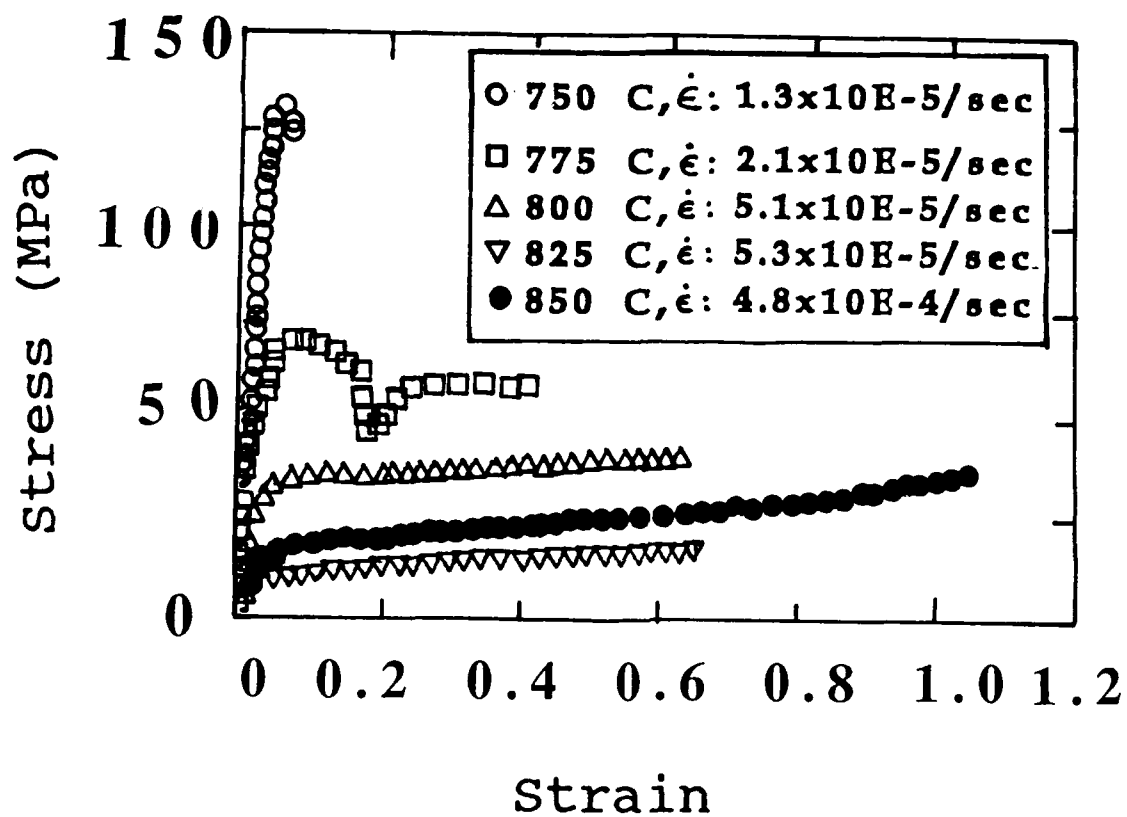


Figure 14. Stress versus strain profiles of 25 vol.% silver / $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ composites processed using 3 micron size commercial powder.

samples did not show any significant change in the grain size. Figure 15 shows typical morphology of the fracture surface of both pure $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ and 25 vol.% silver composites obtained before and after deformation at 850°C . The results shown in Figure 15 indicate that the coarseness in the surface morphology decreased and the grains show some degree of flatness suggesting that the fracture mode has changed from rough intergranular failure to that of a flat intragranular failure. In the case of silver composites, the number of pores on the grain boundary have decreased after deformation.

Both $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ and silver composites that were subjected to the deformation process showed superconducting behavior at the liquid nitrogen temperature (77 K). However, the samples that suffered significant grain flattening during deformation showed a decrease in the absolute value of T_c from 77 K to 70 K. Although we believe that the surface morphology changes are due to changes in the fracture mode from intergranular to intragranular failure, the exact mechanism is still under investigation. The full analysis of the present study will be reported at a later date.

CONCLUSION

From the present investigation the following conclusions can be derived :

1. The addition of alumina to $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ significantly alter the shape and topography of the $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ particles. The addition of silver or silver oxide does not affect the particle morphology.

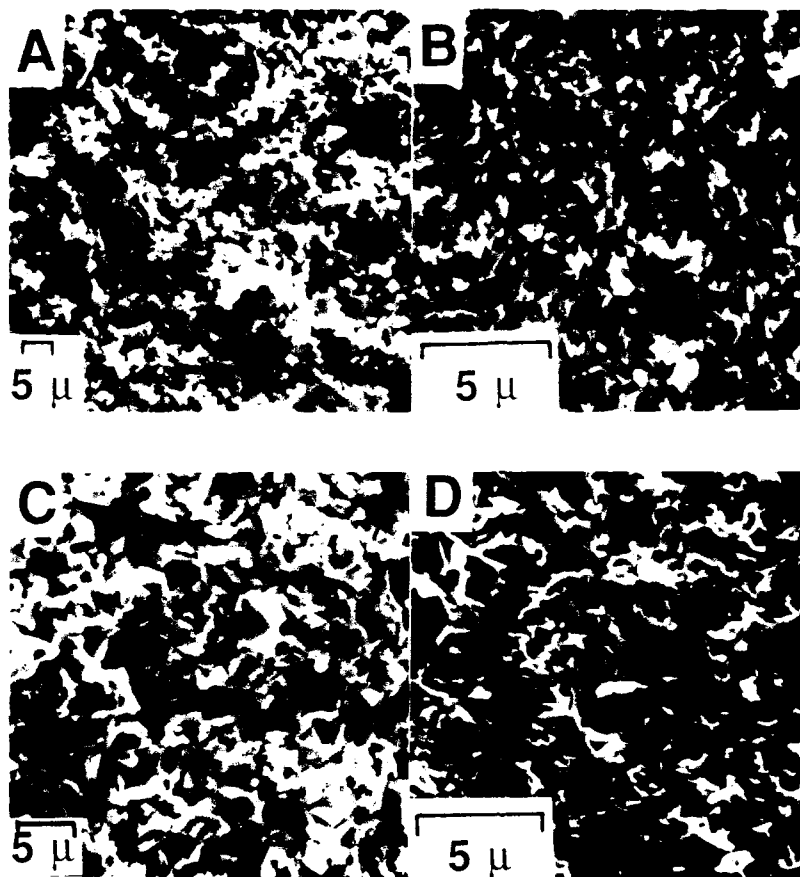


Figure 15. Morphology of the fracture surface of pure $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ [(A) before and (B) after deformation] and 25 vol.% silver composites [(C) before and (D) after deformation]. Deformation temperature 850°C and the deformation strain rate 1.7×10^{-4} per second.

2. The addition of alumina above 5 wt.% reduces the particle size and both silver and silver oxide do not affect the particle size over the concentration range 1 - 25 wt.%.
3. The presence of as little as 1 wt.% alumina tends to lower the superconducting transition temperature (T_C) of a $YBa_2Cu_3O_{6+x}$ composite. Silver or silver oxide tend to improve the T_C of the composite.
4. Alumina composites tend to fail in a brittle fracture mode during deformation, even at 850°C.
5. Pure $YBa_2Cu_3O_{6+x}$ ceramic materials show superplasticity during deformation above 800°C. The maximum deformation that has been achieved in pure $YBa_2Cu_3O_{6+x}$ samples at 850°C was ~ 62 %.
6. Composites containing silver as an additive tend to allow superplastic deformation of up to 110 % without significant degradation of superconducting properties of the composite.

ACKNOWLEDGMENT

The authors would like to thank Prof. Y. T. Chou of the Lehigh University for deformation experiments.

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1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE <div style="text-align: center;">5/15/93</div>	3. REPORT TYPE AND DATES COVERED		
4. TITLE AND SUBTITLE		5. FUNDING NUMBERS		
High Temperature Deformation Behavior of $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ Superconducting Ceramic Materials				
6. AUTHOR(S) <div style="text-align: center;">A. S. Rao, L. F. Aprigliano and O. P. Arora</div>				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Naval Surface Warfare Center Carderock Division Annapolis Detachment Annapolis, MD 21402			8. PERFORMING ORGANIZATION REPORT NUMBER CarderockDiv SSM-61-93/18	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Office of Naval Research CDNSWC - IR Program Annapolis, MD 21402			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unlimited			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Superplastic deformation is being explored as a means to turn the new ceramic superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ into useful Naval hardware. The deformation experiments that were conducted on fine $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ powders suggest that the maximum deformation that can be achieved without the degradation of superconductivity is about 62 % at 850°C. The addition of silver improves the maximum deformation and the addition of alumina has a devastating effect on the deformation mode.				
14. SUBJECT TERMS High Temperature, Deformation, Superplasticity Silver, alumina, Superconductivity			15. NUMBER OF PAGES	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	